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## Growth, optical and XRD studies on pure and doped potassium hydrogen phthalate single crystals

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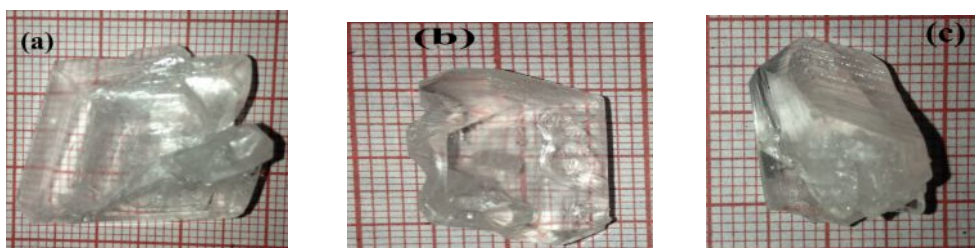
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**Abstract:** Potassium hydrogen phthalate (KHP) is a semi-organic crystal. Pure and amino acids like l-histidine and l-aspartic acid doped single crystals were grown from aqueous solution by slow evaporation technique at ambient temperature. The grown crystals were characterized by FT-IR, UV-Vis spectral studies and powder X-ray diffraction studies. The material of the grown crystals was confirmed by powder X-ray diffraction studies. The powder X-ray diffraction data compares very well with the JCPDS data. The lattice parameters and volume of the pure and doped crystals were calculated. Functional groups and modes of vibrations were identified from FT-IR bands. Optical transmittance was determined for the pure and doped KHP crystals. It reveals that the grown crystal posses as very low absorption in the entire visible and IR region for both pure and doped crystals.  
**Key words:** Semi-organic material; optical transmittance studies; X-ray diffraction; Infrared spectroscopy.

### 1. Introduction and Experimental:

In recent years much attention has been paid to semi-organic NLO materials. The semi-organic materials combine both good qualities of both organic and inorganic materials. Though organic crystals have very large nonlinear susceptibility compared with inorganic crystals, their use is impeded by low optical transparency, poor mechanical strength, low laser damage threshold and inability to produce large crystals[1]. The inorganic NLO materials have excellent mechanical and thermal properties with optical nonlinearities because of the lack of extended  $\pi$ -electron delocalization. Crystals of phthalic acid derivatives are potential candidates for NLO and electro-optic processes. Potassium hydrogen phthalate (KHP) is also called as potassium acid phthalate (KAP) is a semi-organic material. It is one of the well- studied important NLO crystals in the alkali metal acid phthalate (MAP) family[2]. In the present work, the effect of doping on potassium hydrogen phthalate (KHP) by the addition of amino acids like l-histidine and l-aspartic acid were investigated. The grown crystals were characterised by FTIR and UV-vis spectral studies. The PXRD was compared with JCPDS data and the material of the grown crystals was confirmed. The details are presented.

To the saturated solution of KHP, 0.05M l-histidine and l-aspartic acid were added in different beakers with continuous stirring. The solution was filtered and kept in the growth beaker with the top closed with perforated sheet. The crystals were grown by slow evaporation technique at ambient temperature. The crystals were harvested after 25 days. The photograph of the grown crystals was shown in Fig.1.



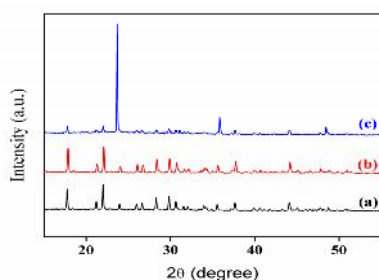
**Fig.1.:** Photographs of grown crystals (a) Pure (b) l-histidine doped (c) l-aspartic acid doped

## 2. Result and discussion:

Powder X-ray pattern of the crystal was recorded using XPERT-PRO X-ray diffractometer with  $\text{CuK}\alpha$  radiation in the wavelength of  $1.5406\text{\AA}$ . To study the linear optical properties, the optical absorption spectrum was measured in the range of 200 to 800nm using UV-2600 UV-vis spectrophotometer (SHIMADZU). The FT-IR spectrum was recorded in the range of 400 to  $4000\text{cm}^{-1}$  employing JASCO FT/IR -4100 spectrometer by KBr pellet method.

### 2.1. Powder X-ray diffraction analysis:

The PXRD pattern of the grown crystals is shown in Fig.2. It is used to confirm the material of the crystal and to calculate the lattice parameters. The PXRD data of pure crystal was compared with the JCPDS data (24-1870 & 31-1855). The cell parameters of pure and doped KHP crystals were calculated using the cell refinement software. There is a slight change in lattice parameters of doped crystals and it is tabulated in Table 1. The pure and doped crystals belong to orthorhombic structure.



**Fig.2.:** Powder X-ray pattern for KHP crystals (a) Pure (b) l-histidine doped (c) l-aspartic acid doped crystals

**Table 1: Lattice parameters of pure and amino acids doped KHP crystals**

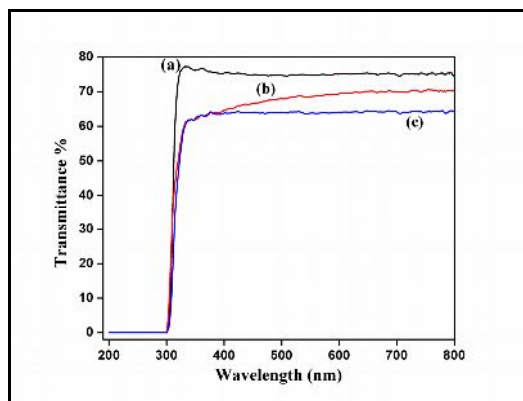
Lattice parameters	JCPDS	Observed	Observed	Observed
	KHP	KHP	0.05M% l-histidine doped KHP	0.05M% l-aspartic acid doped KHP
a( $\text{\AA}$ )	9.605	9.6030	9.5858	9.6083
b( $\text{\AA}$ )	13.331	13.5947	13.3229	13.3190
c( $\text{\AA}$ )	6.473	6.4142	6.4764	6.4811
Volume ( $\text{\AA}^3$ )	828.8305	837.3691	827.1084	829.4113

### 2.2. Optical transmittance studies:

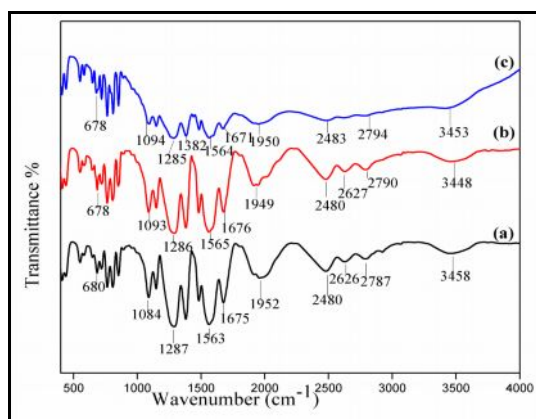
High quality grown crystals with thickness of 2.188, 2.365 and 2.656mm for pure, l-histidine and l-aspartic acid KHP respectively were used for UV-vis spectral studies. The UV spectra of the amino acids doped KHP is shown in Fig.3. All the crystals show 60-80% transmittance in the visible region. The UV cutoff region is 300nm for all the crystals. It is due to n- $\pi$  transition of the carbonyl group of the carboxyl functions.

### 2.3. FTIR Spectral analysis:

The FTIR spectrum of the amino acids doped KHP is shown in Fig.4. In the FT-IR spectra, the presence of amine NH bending peak at  $1565\text{cm}^{-1}$  confirmed the entry of amino acid in the crystal lattice. The C=C stretching is present in  $1675\text{cm}^{-1}$ . Carboxylic acid O-H stretching is present in  $2787\text{cm}^{-1}$ . The vibrational frequency at  $3458\text{cm}^{-1}$  is due to Amine NH stretching.



**Fig.3:UV spectra for KHP crystals (a) Pure (b) l-histidine doped (c) l-aspartic acid doped**



**Fig.4: FTIR spectra for KHP crystals (a) Pure (b) l-histidine doped (c) l-aspartic acid doped**

## 2.4 Conclusion:

Amino acids doped KHP crystals were grown from the aqueous solution using slow evaporation solution growth technique. Powder XRD results confirmed the material of the grown crystals to be KHP. A variation in the lattice parameters were observed on adding the dopants. The FTIR spectral study confirmed the presence of functional group of amino acid in the doped crystal. From the UV-vis spectral study was found to high for pure crystal compared to the doped crystal.

## 3. References:

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